ASCLD/LAB-*International*

ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty – ANNEX A

Details on the NIST 8 Step Process

ASCLD/LAB-*International* is a program of the
American Society of Crime Laboratory Directors / Laboratory Accreditation Board
ASCLD/LAB

Approval Date: August 5, 2011
Approved By: ASCLD/LAB Executive Director
Effective Date: July 1, 2012
ASCLD/LAB Document Control Number: AL-PD-3056 Ver. 1.0

ASCLD/LAB customers should use this document in conjunction with AL-PD-3051 and AL-PD-3055 for conformance with ASCLD/LAB Measurement Uncertainty policy requirements.

NOTE AL-PD-3008 *Estimating Uncertainty of Measurement Policy* and AL-PD-3033 *Updated Approach to Uncertainty of Measurement Requirements are* withdrawn, effective August 5, 2011, and should no longer be used for conformance with ASCLD/LAB measurement uncertainty requirements.

Document History / AL-PD-3056

Date	Version	Description of Activity or Revision	Approved By	Effective Date
July 19, 2011	1.0	Reviewed and adopted by the ASCLD/LAB Board of Directors	Board of Directors	July 1, 2012
August 5, 2011	1.0	Reviewed and approved for distribution by the ASCLD/LAB Executive Director	Executive Director	July 1, 2012

ASCLD/LAB
139 J Technology Drive
Garner, North Carolina 27529
USA
919-773-2600

Purpose

To provide guidance to laboratories that must achieve compliance with the ASCLD/LAB Policy on Measurement Uncertainty[1] as they prepare for and maintain ASCLD/LAB-*International* accreditation.

Scope

This guidance document will cover:

- Details on the NIST 8 Step Process for the estimation of measurement uncertainty
  - This framework can be applied to those tests and calibrations where by policy ASCLD/LAB-*International* has required that the uncertainty be estimated (as demonstrated by the examples provided). The process can also be applied for other tests where statute or customer request requires a laboratory to estimate the uncertainty of a test result.

This guidance document is intended to be used by:

- Testing and Calibration laboratories either currently accredited or seeking accreditation by the ASCLD/LAB-*International* programs,
- Technical assessors for the ASCLD/LAB-*International* programs, and
- Users of ASCLD/LAB-*International* accredited laboratory services.

For ease of use:

❖ Denotes a definition

➢ Denotes a requirement from the cited accreditation requirement document. Accreditation documents include: ISO/IEC 17025:2005,[2] ASCLD/LAB-*International* Supplemental Requirements for both Testing[3] and Calibration[4] programs, ASCLD/LAB Policy on Measurement Uncertainty[1] and ASCLD/LAB Board Interpretations[5]

✓ Denotes a mathematical formula

Introducing NIST's Eight-Step Process for Estimating Uncertainty

The steps in the process are:

- Step 1 • *Specify the Measurement Process*
- Step 2 • *Identify uncertainty sources*
- Step 3 • *Quantify uncertainty sources*
- Step 4 • *Convert factors to standard uncertainties*
- Step 5 • *Calculate combined standard uncertainty*
- Step 6 • *Expand the uncertainty by coverage factor (k)*
- Step 7 • *Evaluate the expanded uncertainty*
- Step 8 • *Report the uncertainty*

The remainder of this document will go through the NIST 8 Step process explaining each step.

Discipline specific examples of estimating the uncertainty of a test or calibration are provided in Annexes B-E. For ease in future document control maintenance, each annex will be issued as a separate document.

Annex B - Testing - Drug Chemistry – Currently under development
Annex C - Testing - Firearms - Currently under development
Annex D - Testing - Toxicology - Currently under development
Annex E - Calibration - Toxicology - Breath Alcohol - Currently under development

1. Specify the measurement process

State the measurand - what is being measured and how. If possible, do this both as a written expression and as a mathematical expression showing the measurement result and the parameters that it depends on.

Being specific is important. Performing the same measurement with different measuring devices or parameters may result in a different estimation of uncertainty.

Expect to come back to Step 1 a number of times throughout the 8 Step process. As you identify uncertainty sources, it may mean revising both your written and mathematical expression. The final description of the process may in fact be a combination of a number of measurement processes. It is easy to see how a mathematical model can become complex and might never be fully delineated.

2. ***Identify uncertainty sources***

- The goal of this step is to comply with ISO/IEC 17025:2005, Clause 5.4.6.3 which states "when estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account ..."

ISO/IEC 17025:2005, Note 1 of section 5.4.6.2 provides additional guidance when it states: "The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

- the requirements of the test method;
- the requirement of the customer;
- the existence of narrow limits on which decisions on conformity to a specification are based."

In the foreword to the second edition of the EURACHEM/CITAC Guide: Quantifying Uncertainty in Analytical Measurement,[6] the author's advice is that "the evaluation of uncertainty requires the analyst [however named] to look closely at all the possible sources of uncertainty. However, although a detailed study of this kind may require a considerable effort, it is essential that the effort expended should not be disproportionate. In practice, a preliminary study will quickly identify the most significant sources of uncertainty ... and the value obtained for the combined uncertainty is almost entirely controlled by the major contributions."

In general, if a mathematical expression has been determined for Step 1, then all parameters in the expression will have one or more components that contribute to the uncertainty.

The *Guide to the expression of uncertainty in measurement* (GUM)[7] advises that it may be useful to consider a test or calibration method as a number of discrete processes and to estimate the uncertainty for each process. This is especially useful if the process is performed in many different test methods.

The key is to realize that you will probably not identify "all" uncertainty components, but you are required to take into account all contributions which are of importance. "Importance" may not be able to be determined until later in the process; therefore, in Step 2, more components may need to be considered and evaluated.

Is there any guidance on where to start to identify uncertainty components?

Yes. Many references on measurement uncertainty also provide a list of possible sources that they recommend be considered. Three references are provided below to allow you to see both the similarities and the differences. The laboratory personnel most familiar with the test or calibration method are generally those who can most easily and completely identify the potential contributions to uncertainty.

The GUM[7] in section 3.3.2 includes:

a. Incomplete definition of the measurand;
b. Imperfect realization of the definition of the measurand;
c. Non-representative sampling – the sample measured may not represent the defined measurand;
d. Inadequate knowledge of the effects of environmental conditions on the measurement or imperfect measurement of environmental conditions;
e. Personal bias in reading analog instruments;
f. Finite instrument resolution or discrimination threshold;

g. Inexact values of measurement standards and reference materials;
h. Inexact values of constants and other parameters obtained from external sources and use in the data-reduction algorithm;
i. Approximations and assumptions incorporated in the measurement method and procedure; and
j. Variations in repeated observations of the measurand under apparently identical conditions. (may be impacted by a-i)

ILAC[8] lists the following Factors Contributing to Uncertainty of Measurement:

Consideration should be given to the different factors which may contribute to the overall uncertainty of a measurement (not all are relevant in all cases). Some examples:

1. definition of the measurand
2. sampling
3. transportation, storage and handling of samples
4. preparation of samples
5. environmental and measurement conditions
6. the personnel carrying out the tests
7. variations in the test procedure
8. the measuring instruments
9. calibration standards or reference materials
10. software and/or, in general, methods associated with the measurement
11. uncertainty arising from correction of the measurement results for systematic effects

A Beginner's Guide to Uncertainty of Measurement[9] lists:

- The measuring instrument
- The item being measured
- The measurement process
- 'Imported' uncertainties
- Operator skill
- Sampling issues
- The environment

Is there a required format for the list of components to uncertainty?

No. You can use any mechanism that works for your laboratory as long as the records are reviewable and maintained. The mechanism can be as simple as a list, a "fishbone" diagram, a "budget" or another choice of the laboratory. The ASCLD/LAB Annexes provide different examples.

An Ishikawa diagram, also referred to as a fishbone diagram or a cause-and-effect diagram is a causal diagram that identifies potential factors causing an overall effect.

Two examples and a blank fishbone diagram are available for your use on the ASCLD/LAB website at www.ascld-lab.org (Choose "Forms" from the main menu). The downloadable diagram can be edited to include more or less "bones" and to reflect the contributions to uncertainty in your process. The "bones" can be typical basic categories in all methods, steps in a specific test or calibration method, or parameters in the mathematical equation stated in Step 1.

A spreadsheet program may be used for the entire 8 Step process. An EXCEL file is available for your use on the ASCLD/LAB website at www.ascld-lab.org (Choose "Forms" from the main menu). The components can be entered into the "budget" directly or after they have been gathered using any other format. Use of the sample EXCEL spreadsheet is not required for accreditation. Components to uncertainty may be gathered using any other format.

Sample of EXCEL spreadsheet:

Item	Uncertainty Component
1	
2	
3	
4	

- Per ISO/IEC 17025:2005, Clause 5.4.7.1, each laboratory will need to ensure that functions of the EXCEL spreadsheet work properly after downloading from the ASCLD/LAB website. Records of this performance verification will need to be maintained and be available for review.

3. Quantify uncertainty sources

In Step 3, depending on the test or calibration method, there may be a blending of the approaches to estimating measurement uncertainty as described in the *ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty - Overview.*[10]

Review the list of uncertainty sources created in Step 2. Which of the components listed, can be quantified by existing data that can be evaluated statistically?

By existing data, do you mean "quality control" data, "method validation" data, or data from "measurement assurance programs"?

Yes. For a new method, the laboratory will have method validation precision data that can be used to quantify uncertainty contributions. Importantly, this is true only if the method validation was structured appropriately to be representative of samples that will be tested or items that will be calibrated. As a part of method validation, it will be necessary to investigate if precision is constant over the range of the method (e.g. extraction efficiency over the expected concentration range, detector response over concentrations, etc.). Method validation should address the impact of the sample matrix, or matrices, on method performance. Once completed, method validation has potentially included the evaluation of a number of significant factors that could contribute to a method's uncertainty.

Once a method has been validated, ISO/IEC 17025:2005 in Clause 5.9.1 states "the laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken..."[2] Quality control data has the potential to provide much of the needed information for estimating uncertainty. Quality control is one aspect of Measurement Assurance.

- **Measurement Assurance** - Practices put in place to monitor a testing or calibration process and/or the reference standards or reference materials used in a process.

The goal of Measurement Assurance is to mimic the test or calibration method. Depending on how well the quality control sample(s) or the measurement assurance sample(s) or check standard(s) mimic the actual test or calibration process will determine how many of the uncertainty components listed in Step 2 will be covered by this precision data.

The Annexes to this document give examples of measurement assurance processes that mimic the test or calibration process.

The GUM has classified uncertainty components that can be evaluated statistically through a series of observations as *Type A*.

- ❖ *Type A* evaluation (of uncertainty) - a method of evaluation of uncertainty by the statistical analysis of a series of observations.[7]

What type of statistical evaluation is performed?

The statistic that is calculated is the standard deviation of the numerical results from the series of measurements gathered through method validation data or ongoing quality control or measurement assurance programs. Standard deviation is a mathematical expression of the variability (dispersion) in this repeatability[11] or reproducibility[12] data – an expression of the average distance from the mean. The mean is the sum of the measurement values divided by the number of measurements. The mean is the best available estimate of the value after a series of independent observations.

Arithmetic Mean:

✓ $$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

The distribution, the spread of a set of data, can take many forms. If an infinite number of measurements were taken and available to calculate the mean, then the mean would approach the 'true value (symbolized as μ)' and the standard deviation would be σ. In practice, the mean and spread are calculated based on a smaller number of measurements and the mean is symbolized as $\bar{x}$ and the standard deviation as *s*.

Standard Deviation (Population):

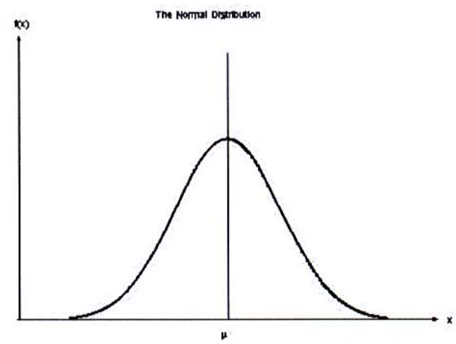
✓ $$\sigma = \sqrt{\frac{\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}{n}}$$

Standard Deviation (Sample):

$$\checkmark \qquad s = \sqrt{\frac{\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}{n - 1}}$$

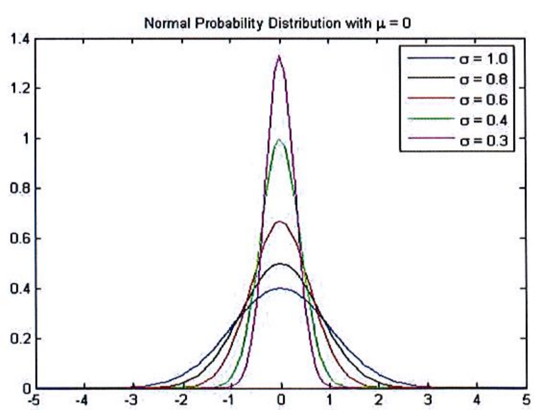
The standard deviation is the variability due to random effects from various sources that affect the measurement result. If the variation is due to random effects, with a large enough number of measurements the data will approximate a normal distribution. In a normal distribution (Figure 1), the majority of the measurement results center around the mean and the shape of the curve is symmetrical about the mean. It is less likely to observe data in the margins of the distribution.

Normal Distribution (Gaussian distribution)
Figure 1



Source: http://www.itl.nist.gov/div898/handbook/pmc/section5/pmc51.htm

Figure 2 shows five populations of data with the same mean but different standard deviations. Greater variability (the width of the bell shaped curve gets broader) translates to a larger standard deviation.

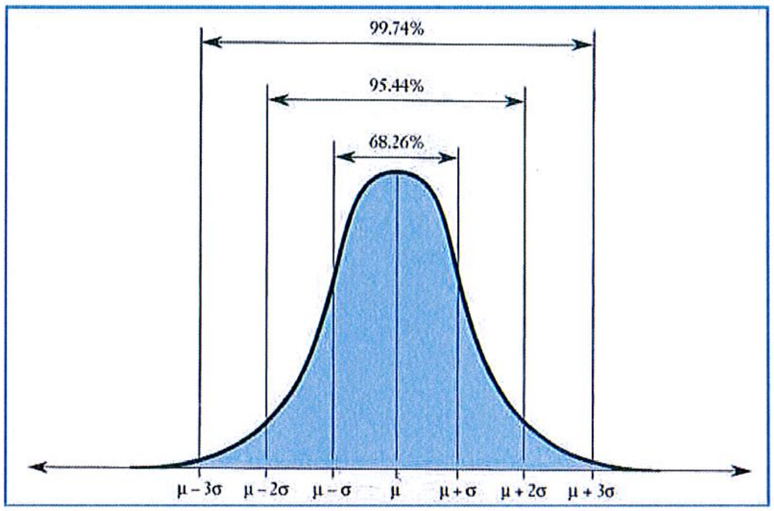


Normal Distribution (Gaussian distribution)

Figure 2: Five populations with the same mean but different standard deviations

If your data follows a normal distribution, then the probability that the value of your next measurement result of the same measurand, under similar operating conditions, would be within one standard deviation of the mean is approximately 68%. The probability that the value would be within 2 standard deviations is approximately 95% and the probability that the value would be within 3 standard deviations of the mean is approximately 99 %.(Figure 3)

Normal Distribution (Gaussian distribution)
Figure 3: Figure 1 graph with probabilities added



After identifying contributions to uncertainty for which the laboratory has experimental data that can be statistically evaluated (*Type A*), there are still components to uncertainty identified in Step 2 that have not been quantified or evaluated. What does the laboratory do with these other components?

Even when quality assurance or measurement assurance processes closely mimic the test or calibration method, there will be uncertainty attributed from other sources. These may include, but are not limited to, reference materials, reference standards or, at times, an environmental condition that is not covered by the *Type A* data.

How do I evaluate these types of uncertainty components?

The evaluation of the remaining uncertainty components is performed by a different classification of evaluation. The GUM has classified this type of evaluation as *Type B.*

- *Type B* evaluation (of uncertainty) - a method of evaluation of uncertainty by means other than the statistical analysis of a series of observations.

If the laboratory determines that an uncertainty component cannot be evaluated statistically, or that a statistical evaluation would be impractical, or that a statistical evaluation may be unnecessary, this demonstrates that the uncertainty can be categorized as a *Type B* uncertainty.